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मानक

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IS 1832 (1978): Specification for Malathion, Technical [FAD
1: Pesticides and Pesticides Residue Analysis]



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“Knowledge is such a treasure which cannot be stolen”

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IS : 1832 - 1978

(Reaffirmed 2012)

Indian Standard
**SPECIFICATION FOR
MALATHION, TECHNICAL**
(First Revision)

Fourth Reprint JANUARY 2001

UDC 632.951 MAL

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**BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002**

AMENDMENT NO. 1 JANUARY 1980
TO

IS : 1832-1978 SPECIFICATION FOR
MALATHION, TECHNICAL

(*First Revision*)

Alterations

(*Pages 6, 7, 8 and 9, clauses A-1 to A-1.6.2*) — Substitute the following for the existing clauses:

'A-1. PRINCIPLE OF METHOD

A-1.1 Malathion, *S*-1, 2-di (ethoxycarbonyl) ethyl *O*, *O*-dimethyl phosphorodithioate, is decomposed by alkali in ethyl alcohol or methyl alcohol to sodium *O*, *O*-dimethyl dithiophosphate sodium fumarate, and ethyl alcohol. The sodium *O*, *O*-dimethyl dithiophosphate is then converted to the cupric complex which is soluble in carbon tetrachloride with the formation of an intense yellow colour. The colour intensity is proportional to the concentration of *O*, *O*-dimethyl dithiophosphoric acid and is measured colorimetrically at 420 nm, the absorption peak. The corresponding amount of the insecticide is derived by comparison with a standard prepared with a known amount of the pure insecticide, which is simultaneously carried through the procedure.

A-1.1.1 In this method, ferric reagent is added to oxidize materials which would reduce cupric ions to cuprous ions; with *O*, *O*-dimethyl dithiophonic acid (DMTA), cuprous ions form a colourless complex which is apparently more stable than the yellow cupric complex.

A-1.2 Apparatus

A-1.2.1 *Spectrophotometer or Photoelectric Colorimeter* — with a blue filter (420 nm) and having 1-cm absorption cell.

A-1.3 Reagents

A-1.3.1 *Malathion*

A-1.3.2 *Acetonitrile* — boiling range 80 to 82°C.

A-1.3.3 *Ethyl Alcohol* — anhydrous, alternatively methyl alcohol anhydrous may be used.

A-1.3.4 Carbon Tetrachloride

A-1.3.5 Sodium Hydroxide Solution — 0.5 N. Dissolve 4.0 g of sodium hydroxide in 200 ml of water to get a clear solution.

A-1.3.6 Ferric Reagent

A-1.3.6.1 Stock Solution — Dissolve 4.0 g of ferric chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) and 160 ml of concentrated hydrochloric acid in water and dilute to 500 ml.

A-1.3.6.2 Dilute solution — Dilute 25 ml of the stock solution with water to 1 000 ml.

A-1.3.7 Copper Sulphate Solution — 1.5 percent (m/v). Prepare by dissolving 1.5 g of copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in 100 ml of water.

A-1.4 Preparation of Standard and Sample Solutions

A-1.4.0 It is very important in preparing the *Standard* and *Sample Solutions* that volume fluctuations with changing temperatures are avoided. This is accomplished by bringing each solution to the volume mark just before proceeding to the next step in the procedure, as indicated or preferably analysis shall be carried in a room maintained at constant temperature.

A-1.4.1 Preparation of Standard Solution

A-1.4.1.1 Accurately weigh into a 5-ml or 10-ml beaker, 0.100 ± 0.020 g of pure malathion. Quantitatively transfer it to a 100-ml volumetric flask with anhydrous ethyl alcohol or anhydrous methyl alcohol. Then dilute with ethyl alcohol or methyl alcohol to approximately 5 ml below the volume mark. Allow the solution to come to a selected temperature. Record this temperature. Dilute the solution with corresponding alcohol to the mark and mix well. Call this as *Standard Solution A*.

NOTE — *Standard Solution A* may be retained for daily use up to two weeks. It should be preferably kept at low temperature. It is very important, however, that it is adjusted to the temperature recorded above before aliquots are withdrawn.

A-1.4.1.2 Transfer a 10 ml aliquot of *Standard Solution A* into a 100-ml graduated flask. Add 1 ml acetonitrile and dilute with corresponding alcohol to approximately 5 ml below the volume mark. Retain the solution until *Sample Solution* (see A-1.4.2) is prepared and is ready for final dilution. At that time, dilute with corresponding alcohol to the mark and mix well. Call this as *Standard Solution*.

A-1.4.2 Preparation of Sample Solution

A-1.4.2.1 Accurately weigh a quantity of the sample containing 0.100 ± 0.020 g of malathion and quantitatively transfer it to a 100-ml

volumetric flask with anhydrous ethyl alcohol or anhydrous methyl alcohol and make up to the mark with corresponding alcohol at selected temperature (see A-1.4.1.1). Call this as *Sample Solution A*.

A-1.4.2.2 Immediately transfer a 10 ml aliquot of *Sample Solution A* into a 100-ml graduated flask. Add 1 ml acetonitrile and dilute it to mark with corresponding alcohol at selected temperature (see A-1.4.1.1) at the time of final dilution. Call this as *Sample Solution*.

NOTE — If more than one sample solutions are being prepared for assay, then all *Sample Solutions* and the *Standard Solutions* should be diluted to approximately 5 ml below the volume mark as prepared. All shall be diluted to the volume mark at the same time.

A-1.5 Procedure

A-1.5.0 To obtain accurate results, it is important that the analysis of *Standard* and *Sample Solutions* be carried simultaneously through the procedure given in A-1.5.1 to A-1.5.4 without interruption.

A-1.5.1 Transfer a 15 ml aliquot of *Standard Solution* and 15 ml of *Sample Solution* to two different 250-ml separating funnels. Add 2 ml of sodium hydroxide solution and mix well by swirling gently (do not shake) for 5 to 10 seconds. Allow the solution to stand for exactly 2 minutes.

A-1.5.2 Immediately add 75 ml of ferric reagent and mix well by swirling for 10 seconds. Allow the solution to stand for exactly 5 minutes. During this 5 minutes period, fill two 50-ml volumetric flasks to the mark with carbon tetrachloride and after the lapse of 5 minutes simultaneously transfer the carbon tetrachloride from each volumetric flask to the respective separating funnels. Allow the flask to drain for one minute.

NOTE — From this point onward the *Sample Solution* and *Standard Solution* should be handled separately. Carry each one through the next step without interruption.

A-1.5.3 Add 2 ml of copper reagent. Start shaking the separating funnel immediately after addition of copper reagent for exactly one minute. The copper complex of DMTA is unstable in the aqueous phase. The copper reagent should be added with a fast delivery pipette and the shaking should be started with practically no time lapse. Allow the phase to separate. Allow some solvent phase to rinse the stem of the separating funnel, then run the clear solvent phase directly into the cell.

A-1.5.4 Measure the absorbance of the yellow solution within 2 minutes at 420 nm using carbon tetrachloride as the blank.

NOTE — Absorbance measurements of both *Standard* and *Sample Solutions* should be made at the same fixed time after the end of the shaking period. The yellow colour of the copper complex in carbon tetrachloride is unstable, hence the colour should be read within 2 minutes after extraction. During this time the absorbance reading drops very slowly.

A-1.6 Calculation

A-1.6.1 The absorbance measurements of the *Standard* and *Sample Solutions* should agree within ± 10 percent of each other.

A-1.6.2 Malathion content, percent by mass = $\frac{A}{B} \times \frac{m}{M} \times f$

where

A = absorbance of *Sample Solution* at 420 nm,

m = mass in g of pure malathion taken for the preparation of *Standard Solution*,

f = percentage purity of the malathion used for the preparation of *Standard Solution*,

B = absorbance of *Standard Solution* at 420 nm, and

M = mass in g of the material taken for the preparation of the *Sample Solution*.'

(Page 10, clause A-2.3.4) — Substitute the following for the existing clause:

'A-2.3.4 Alcoholic Sodium Hydroxide Solution — 1 N.'

(Page 12, clauses B-3 to B-3.2) — Substitute the following for the existing clauses:

'B-3. DETERMINATION OF PURITY

B-3.1 Preparation of Perchloric Acid — Dissolve 73 g of 70 percent perchloric acid with 124.1 g of acetic anhydride and dilute to 5 litres with glacial acetic acid.

NOTE — If the acetic acid used is not anhydrous, a corresponding larger volume of acetic anhydride will be required, equivalent to the total water content of the other two reagents.

B-3.2 Standardization of Perchloric Acid — Dissolve 0.2 g of anhydrous sodium carbonate, accurately weighed, in 50-ml of glacial acetic acid and titrate this solution with the perchloric acid solution, using 1 percent α -naphtholbenzein solution in benzene as indicator. 0.200 g of sodium carbonate corresponds to 37.73 ml of 0.1 *N* perchloric acid.

B-3.3 Determination of Purity of the Sample — Dissolve 0.300 g of the salt in 25 ml of glacial acetic acid and titrate against 0.1 *N* perchloric acid, potentiometrically (see B-3.2).

B-3.4 Calculate the percentage purity of the salt. The purity obtained by the method is greater than 99 percent.'

(AFCD 6)

AMENDMENT NO. 2 OCTOBER 1980

TO

**IS : 1832 - 1978 SPECIFICATION FOR
MALATHION, TECHNICAL**

(*First Revision*)

Alterations

[*Page 7, clause A-1.3.1 (see also Amendment No. 1)*] — Substitute the following for the existing clause:

' A-1.3.1 Malathion — pure of + 98 percent purity by GLC or colorimetric method.'

[*Page 9, clause A-1.6.2 (see also Amendment No. 1)*] — Substitute the following for the existing formula:

'A-1.6.2 Malathion content, percent by mass,
if GLC reference material
(*see A-1.3.1*) has been used $= \frac{A}{B} \times \frac{m}{M} \times f \times 1.03$

OR

Malathion content, percent by mass,
if colorimetric reference material
(*see A-1.3.1*) has been used $= \frac{A}{B} \times \frac{m}{M} \times f$

(AFGDC 6)

Indian Standard

SPECIFICATION FOR MALATHION, TECHNICAL

(First Revision)

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Indian Standard

SPECIFICATION FOR MALATHION, TECHNICAL

(*First Revision*)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 20 March 1978, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 This standard was first issued in 1961. In this revision an additional method for estimation of malathion content has been given. Other changes pertain to the requirements of packaging, marking and sampling. Opportunity has been taken to refer to IS : 6940-1973* to bring uniformity in testing procedure for the various requirements given in the standard.

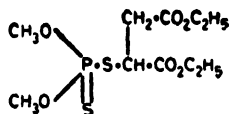
0.3 Malathion, technical is used in the preparation of formulations required for the control of agricultural and public health pests.

0.4 Malathion is the accepted common name by the International Organization for Standardization (ISO) for *S*-1, 2-di (ethoxycarbonyl) ethyl *O* *O*-dimethyl phosphorodithioate. The empirical and structural formulae and the molecular mass of malathion are as indicated below:

Empirical Formula



Structural Formula



Molecular Mass

330.4

*Methods of tests for pesticides and their formulations.

IS : 1832 - 1978

0.5 In the preparation of this standard, due consideration has been given to the provisions of the Insecticides Act, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for malathion, technical, employed in the preparation of insecticidal formulations.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of a colourless to amber liquid free from extraneous impurities, sediments or added modifying agents.

2.2 The material shall also comply with the requirements given in Table 1.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed according to IS : 8190 (Part II)-1976†.

3.2 Marking — The containers shall bear legibly and indelibly the following information in addition to the provisions of the Insecticides Act and Rules:

- a) Name of the material;
- b) Name of manufacturer;
- c) Date of manufacture;
- d) Batch number;
- e) Net mass of contents;
- f) Malathion content, percent (*m/m*); and
- g) The cautionary notice as worded in the Insecticides Act and Rules.

*Rules for rounding off numerical values (*revised*).

†Requirements for packing of pesticides: Part II Liquid pesticides.

TABLE 1 REQUIREMENTS FOR MALATHION, TECHNICAL
(Clauses 2.2 and 5.1)

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO	
			Appendix of this Standard	Cl No. of IS : 6940-1973*
(1)	(2)	(3)	(4)	(5)
i)	Malathion content, percent by mass, <i>Min</i>	95.0	A	—
ii)	Moisture content, percent by mass, <i>Max</i>	0.1	—	4.1
iii)	Acidity (as H_2SO_4), percent by mass, <i>Max</i>	0.4	—	11.3.2
iv)	Material insoluble in acetone, percent by mass, <i>Max</i>	0.5	—	9
v)	Specific gravity at 25/25°C, <i>Min</i>	1.23	—	5

*Methods of tests for pesticides and their formulations.

3.2.1 The product may also be marked with Standard mark.

3.2.1.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in the 'Indian Standard Methods for sampling of pesticides and their formulations' (*under preparation*).

NOTE — Till such time the standard under preparation is published, the matter shall be as agreed to between the concerned parties.

5. TESTS

5.1 Tests shall be carried out as prescribed in col 4 and 5 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

[*Table 1, Item (i)*]

DETERMINATION OF MALATHION CONTENT

A-0. GENERAL

A-0.1 For the determination of malathion content, two methods, one using the pure Standard malathion (*see* A-1) and the other using Potassium salt of *O,O* dimethyl phosphorodithioic acid (*see* A-2) have been specified. Either one of the two methods may be used for determination of malathion content. However, in case of dispute, the method employing use of pure Standard malathion (*see* A-1) will be referee method.

A-1. PRINCIPLE OF METHOD

A-1.1 Malathion, *S*-1, 2-di (ethoxycarbonyl) ethyl *O,O*-dimethyl phosphorodithioate, is decomposed by alkali in ethyl alcohol to sodium *O,O*-dimethyl dithiophosphate, sodium fumarate, and ethyl alcohol. The sodium *O,O*-dimethyl dithiophosphate is then converted to the cupric complex which is soluble in carbon tetrachloride with the formation of an intense yellow colour. The colour intensity is proportional to the concentration of *O,O*-dimethyl dithiophosphoric acid and is measured colorimetrically at 420 nm, the absorption peak. The corresponding amount of the insecticide is derived by comparison with a standard prepared with a known amount of the pure insecticide which is simultaneously carried through the procedure.

A-1.1.1 In this method, ferric reagent is added to oxidize materials which would reduce cupric ions to cuprous ions; with dithiophosphoric acid, cuprous ions form a colourless complex which is apparently more stable than the yellow cupric complex.

A-1.2 Apparatus

A-1.2.1 Spectrophotometer or Photoelectric Colorimeter — with a blue filter (420 nm) and having 1-cm absorption cells.

*Specification for water for general laboratory use (*second revision*).

A-1.3 Reagents**A-1.3.1 Malathion**

A-1.3.2 Acetonitrile — boiling range 80 to 82°C.

A-1.3.3 Ethyl Alcohol — anhydrous.

A-1.3.4 Carbon Tetrachloride

A-1.3.5 Sodium Hydroxide Solution — 0.5 N. Dissolve 4.0 g of sodium hydroxide in 200 ml of water.

A-1.3.6 Ferric Reagent

A-1.3.6.1 Stock solution — Dissolve 4.0 g of ferric chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) and 160 ml of concentrated hydrochloric acid in water and dilute to 500 ml.

A-1.3.6.2 Dilute solution — Dilute 25 ml of the stock solution with water to 1 000 ml.

A-1.3.7 Copper Sulphate Solution — 1.5 percent (m/v). Prepare by dissolving 1.5 g of copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in 100 ml of water.

A-1.4 Preparation of Standard and Sample Solutions

A-1.4.0 It is very important in preparing the *Standard* and *Sample solutions* that volume fluctuations with changing temperatures are avoided. This is accomplished by bringing each solution to the volume mark just before proceeding to the next step in the procedure, as indicated.

A-1.4.1 Preparation of Standard Solution

A-1.4.1.1 Accurately weigh into a 5-ml or 10-ml beaker, 0.100 ± 0.020 g of pure malathion. Quantitatively transfer it to a 100-ml graduated flask with ethyl alcohol. Then dilute with ethyl alcohol to approximately 5 ml below the volume mark. Allow the solution to come to a selected temperature. Record this temperature. Dilute the solution with ethyl alcohol to the mark and mix well. Call this as *Standard Solution A*.

NOTE — *Standard Solution A* may be retained for daily use up to two weeks. It is very important, however, that it be adjusted to the temperature recorded above before aliquots are withdrawn.

A-1.4.1.2 Transfer a 15 ml aliquot of *Standard Solution A* into a 250-ml graduated flask. Add 2.5 ml acetonitrile and dilute with ethyl alcohol to approximately 5 ml below the volume mark. Retain the solution until *Sample Solution* (see A-1.4.2) is prepared and is ready for final dilution. At that time, dilute with ethyl alcohol to the mark and mix well. Call this as *Standard Solution*.

A-1.4.2 Preparation of Sample Solutions

A-1.4.2.1 Accurately weigh a quantity of the sample containing 0.300 ± 0.020 g of malathion and quantitatively transfer to a 50-ml bottle and then add 50 ml of acetonitrile. Close the bottle tightly with a screw cap having a polyethylene liner and shake it vigorously for 2 to 3 minutes either manually or on a reciprocating shaker. Then allow the solids to settle for 3 to 5 minutes, if necessary, centrifuge for 2 to 3 minutes at 1 500 to 2 000 rev/min. Call the supernatant liquid as *Sample Solution A*.

A-1.4.2.2 Immediately transfer a 25 ml aliquot of *Sample Solution A* to a 250-ml graduated flask. Dilute with ethyl alcohol to the mark and mix well. Call this as *Sample Solution B*. Then immediately, transfer a 25 ml aliquot of *Sample Solution B* to a 250-ml graduated flask. Dilute with ethyl alcohol to the mark and mix well. Call this as *Sample Solution*. At this time, also dilute the *Standard Solution*.

NOTE — If more than one sample solution is being prepared for assay, then all *Sample Solutions* and the *Standard Solution* should be diluted to approximately 5 ml below the volume mark as prepared. All should be diluted to the volume mark at the same time.

A-1.5 Procedure

A-1.5.0 To obtain accurate results, it is important that the analysis of *Standard* and *Sample Solutions* be carried simultaneously through the procedure given in A-1.5.1 to A-1.5.4 without interruption.

A-1.5.1 Transfer a 25 ml aliquot of *Standard Solution* and one *Sample Solution* to two different 250-ml separating funnels. Add 2 ml of sodium hydroxide solution and mix well by swirling gently (do not shake) for 5 to 10 seconds. Allow the solution to stand for exactly 2 minutes.

A-1.5.2 Immediately add 75 ml of ferric reagent and mix well by swirling for 10 seconds. Allow the solution to stand for exactly 5 minutes. During this 5 minutes period, fill two 50-ml graduated flasks to the mark with carbon tetrachloride and after the lapse of 5 minutes simultaneously transfer the carbon tetrachloride from each graduated flask to the respective separating funnels. Allow the flask to drain for one minute.

NOTE — From this point onward the *Sample Solution* and *Standard Solution* should be handled separately. Carry each one through the next step without interruption.

A-1.5.3 Add 2 ml of copper reagent. Start shaking the separating funnel immediately after addition of copper reagent for exactly one minute. The malathion-copper complex is unstable in the aqueous phase. The copper reagent should be added with a fast delivery pipette and the shaking should be started with practically no time lapse. Allow

the phases to separate. As soon as separation occurs, discard the aqueous phase. Allow some solvent phase to rinse the stem of the separating funnel, then run the clear solvent phase into a small beaker for transferring to an absorption cell or run the clear solvent phase directly into the cell.

A-1.5.4 Measure the absorbance of the yellow solution within 5 minutes at 420 nm using carbon tetrachloride as the blank.

NOTE — Absorbance measurements of both *Standard* and *Sample Solutions* should be made at the same fixed time after the end of the shaking period. The yellow colour of the copper complex in carbon tetrachloride is unstable, hence the colour should be read within 5 minutes after extraction. During this time the absorbance reading drops very slowly.

A-1.6 Calculation

A-1.6.1 The absorbance measurements of the *Standard* and *Sample Solutions* should agree within ± 10 percent of each other.

A-1.6.2 Malathion content, percent by mass = $\frac{A}{B} \times \frac{m}{M} \times f \times 3.0$

where

A = absorbance of *Sample Solution* at 420 nm,

m = mass in g of pure malathion taken for the preparation of *Standard Solution*,

f = percentage purity of the malathion used for the preparation of *Standard Solution*,

B = absorbance of *Standard Solution* at 420 nm, and

M = mass in g of the material taken for the preparation of the *Sample Solution*.

A-2. PRINCIPLE OF METHOD

A-2.1 The sample is hydrolysed quantitatively with ethanolic alkali in an anhydrous medium to give the sodium salt of *O,O*-dimethyl phosphorodithioic acid. The salt is then dissolved in ethyl acetate in which the excess alkali is insoluble. An aliquot is diluted with cyclohexane and copper naphthenate is added. The copper (II) ion reacts with the *O,O*-dimethyl phosphorodithioate ion to form a yellow-coloured complex. The intensity of the colour is proportional to the concentration of *O,O*-dimethyl phosphorodithioate ion present and is measured colorimetrically at 420 nm. The amount of diethyl mercaptosuccinate *S*-ester with *O,O*-dimethyl phosphorodithioate is then determined by comparing the observed absorbance measurements with a calibration graph relating absorbance to various concentration of primary standard potassium salt of *O,O*-dimethyl phosphorodithioic acid complexed with copper (II) ion in a similar manner. Water and free mineral acids are excluded from the reagents to prevent decomposition of the copper complex.

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A-2.2 Apparatus

A-2.2.1 Spectrophotometer or Photoelectric Colorimeter — with blue filter (420 nm).

A-2.3 Reagents

A-2.3.1 Potassium Salt of *O,O*-Dimethyl Phosphorodithioic Acid — Prepare the pure salt according to directions given in Appendix B.

A-2.3.2 Cyclohexane — 98 percent passed through a column of activated silica gel to remove polar materials.

A-2.3.3 Ethyl Acetate

A-2.3.4 Alcoholic Sodium Hydroxide Solution — 0.1 N in ethanol. Prepare by dissolving 4 g of carbonate free NaOH in 100 ml of absolute alcohol.

A-2.3.5 Copper Reagent — Dissolve 420 ± 10 mg of copper (II) naphthenate in 100 ml of cyclohexane.

A-2.3.6 Acetone

A-2.4 Procedure

A-2.4.1 Preparation of the Standard Curve

A-2.4.1.1 Weigh accurately 0.08-0.12 g of pure potassium salt of *O,O*-dimethyl phosphorodithioic acid into a 50-ml volumetric flask, dilute to volume with acetone and mix.

A-2.4.1.2 Pipette 5 ml of this solution into a 50-ml volumetric flask, add 2 ml of sodium hydroxide solution, make up to the mark with ethyl acetate, mix and allow to stand for 10 minutes.

A-2.4.1.3 Transfer portions to the 15-ml stoppered centrifuge tubes and centrifuge for 5 minutes. Pipette 3, 4, 5 and 6 ml of the centrifuged ethyl acetate solution into separate 50-ml volumetric flasks taking care not to transfer any of the precipitated sodium acetate. Add approximately 35 ml of cyclohexane followed immediately by 2 ml of copper reagent. Mix and make each up to the mark with cyclohexane.

A-2.4.1.4 Measure the absorbance of each solution at the wavelength maximum near 420 nm within 2 minutes after adding the copper reagent, using a reference solution carried through the same steps but containing no standard salt.

A-2.4.1.5 Prepare a calibration graph by plotting the absorbances observed for the four solutions against their *O,O*-dimethyl phosphorodithioic acid, potassium salt content in micrograms per ml.

A-2.4.2 Preparation of the Sample

A-2.4.2.1 Weigh accurately a sample containing 0.15 ± 0.05 g of malathion into a 50-ml volumetric flask, dissolve, and make up to the mark with cyclohexane.

A-2.4.2.2 Pipette a 5 ml aliquot into another 50-ml volumetric flask, add 2 ml of sodium hydroxide solution, mix, and allow to stand for 10 minutes. Make up to the mark with ethyl acetate, mix, and allow to stand for 10 minutes. Transfer approximately 10 ml of the solution to a 15-ml stoppered centrifuge tube and centrifuge for 5 minutes.

A-2.4.3 Analysis of the Material

A-2.4.3.1 Pipette 5 ml of the centrifuged ethyl acetate solution of the sample into a 50-ml volumetric flask taking care not to transfer any of the precipitated sodium acetate. Add approximately 35 ml of cyclohexane followed immediately by 2 ml of copper reagent. Mix and make up to the mark with cyclohexane.

A-2.4.3.2 Measure the absorbance at a wavelength maximum near 420 nm 2 minutes after adding the copper reagent, using a reference solution carried through the same steps but containing no malathion.

A-2.4.3.3 Refer to the calibration graph and determine the equivalent concentration of *O,O*-dimethyl phosphorodithioic acid, potassium salt in the solution in micrograms per ml.

A-2.5 Calculation

$$\text{Malathion content, percent by mass} = \frac{a \times 1.683}{m \times 2}$$

where

a = equivalent concentration ($\mu\text{g/ml}$) of *O,O*-dimethyl phosphorodithioic acid, potassium salt in the unknown solution, determined from the calibration graph; and

m = mass in g of the sample taken.

NOTE — Since volume changes of nonaqueous solutions with temperature are appreciable, the analysis of the sample should be carried out, as far as possible, at the temperature at which the calibration graph was prepared.

APPENDIX B

(Clause A-2.3.1)

PREPARATION OF THE POTASSIUM SALT OF O,O-DIMETHYL PHOSPHORODITHIOIC ACID

B-1. REAGENTS

B-1.1 Phosphorus Pentasulphide — A.R.

B-1.2 Diethyl Ether

B-1.3 Methanol

B-1.4 Ethanol — anhydrous.

B-1.5 Benzene — free of thiophene.

B-1.6 Methanolic Potassium Hydroxide — 3 N. Dissolve 168 g of KOH in 1 litre of methanol.

B-2. PROCEDURE

B-2.1 Make a suspension of 222 g of phosphorus pentasulphide in 100 ml of benzene and add, under cooling, 128 g of methanol. After the reaction has finished, neutralize to pH not below 4, with 600-700 ml of 3 N methanolic potassium hydroxide, using a pH meter and cooling in ice so that the temperature does not exceed 20°C.

B-2.2 Filter the reaction mixture and add 1 000 ml of diethyl ether to precipitate the potassium salt. Filter off the precipitate, wash with diethyl ether, and dry. Recrystallize once from methanol and twice from ethanol, preparing saturated solutions by short boiling and precipitating by cooling to 0°C. Finally, dry the salt in a vacuum desiccator.

B-3. DETERMINATION OF PURITY

B-3.1 Dissolve 0.300 g of the salt in 25 ml of glacial acetic acid and titrate with 0.1 N perchloric acid; 1.00 ml of 0.1 N perchloric acid is equivalent to 0.019 3 g of the pure potassium salt.

B-3.2 Calculate the percentage purity. The purity obtained by this method is greater than 99 percent.

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